

ACTIVELY-COOLED FIBER-REINFORCED CERAMIC MATRIX  
COMPOSITE ROCKET PROPULSION THRUST CHAMBER  
AND METHOD OF PRODUCING THE SAME

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CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of Application No. 09/772,108 filed January 25, 2001.

FIELD OF THE INVENTION

The present invention relates to actively-cooled tubular shell structures for high-temperature applications having a plurality of cooling channels formed between the inner wall and outer wall of the structure wherein the structure is fabricated from fiber-reinforced ceramic matrix composite materials and methods of producing same.

BACKGROUND OF THE INVENTION

Performance of advanced chemical rocket technology is limited, for the most part, by the availability of high-temperature structural engineering materials. Greater performance and efficiency in liquid rocket propulsion systems can be gained by operating at higher combustion temperatures and higher working pressures resulting from highly energetic propellant mixture ratios (oxidizer/fuel). However, propellant mixtures are typically "tamed" to off-optimum conditions in order not to exceed the temperature limitations of the thrust chamber and nozzle materials.

A considerable amount of heat is transferred in all designs of rocket engines. The principle objective of high-temperature rocket design is to safely limit the heat transfer to the materials in critical hot sections such as the injector, combustion chamber, throat, and nozzle. A

failure would impair the satisfactory operation of the rocket propulsion system or the flight vehicle being propelled. The walls have to be maintained sufficiently cool so that wall temperatures do not exceed their safe allowable operating limit. Erosion, usually the result of combined oxidation and chemical interaction with the hot combustion gases, should not damage the walls, and the walls should be capable of withstanding the extreme thermal shock caused by the sudden onset of a high heat flux from combustion ignition. The materials comprising the thrust chamber devices must also be capable of resisting the thermal stresses induced by the heat transfer and thermal gradients.

There are two general cooling methods commonly used today in the design of liquid propellant rocket engine devices. Those devices that reach thermal equilibrium during operation typically operate for long durations and are usually either actively-cooled (e.g., bipropellants) or radiation cooled (e.g., monopropellants).

Actively-cooled liquid propellant thrust chambers have provisions for cooling some or all of the components in contact with the hot combustion gases, such as the chamber walls, nozzle walls and injector faces. A cooling jacket or cooling coil often consists of separate inner and outer walls or a bundled assembly of continuous, contoured tubes. The inner wall confines the combustion gases, and the space between the inner and outer walls serves as the coolant passage.

The axial or helical passages in liquid propellant rocket thrust chambers are often of complex cross-section. The nozzle throat region is usually the location that sustains the greatest heat transfer intensity and is therefore the most difficult to cool. For this reason the cooling jacket is often designed so that the coolant velocity is highest at the critical regions by restricting the coolant passage cross-section and so that the coolant enters the jacket at or near the nozzle.

Regenerative cooling is a form of active cooling and is used for engines where one of the propellant constituents is circulated through cooling passages around the thrust chamber prior to injection and burning of the propellant in the combustion chamber. Regenerative cooling in bipropellant engines uses either the fuel or oxidizer as the cooling fluid. Therefore, the thermal energy absorbed by the coolant is not wasted as it augments the initial energy content of the propellant prior to injection, thereby increasing the exhaust velocity and propulsive performance.

Radiation cooling is typically used in monopropellant thrust chambers, some gas generators and for nozzle exhaust sections. Radiation cooling is a simple, lightweight cooling method, which is commonly employed in low-temperature rocket engines, such as hydrazine (monopropellant) spacecraft maneuver and attitude control systems, where the maximum chamber temperature is only about 650°C. Refractory metals such as molybdenum, tantalum, tungsten, and niobium have been used in radiation cooled thrust chambers requiring increased operating temperatures up to 1650°C. Refractory metals are, however, difficult to fabricate and some suffer from hydrogen embrittlement degradation; others oxidize readily and thus require protective surface coatings to function reliably; all are weight inefficient due to their very high specific gravities. Radiation cooled thrust chambers generally have to protrude beyond the outer skin of the flight vehicle to permit satisfactory radiative heat rejection.

In general, ceramics have superior high-temperature strength and stiffness, and lower density than metallic materials. The principal disadvantages of ceramics as structural materials are their low failure strain, low fracture toughness and catastrophic brittle failure characteristics. Because of these inherent limitations, monolithic ceramics lack the properties of reliability and durability that are necessary for structural design acceptance. However, the emerging technology of fiber-reinforced ceramics, or ceramic matrix composites is one promising solution for

overcoming the reliability and durability problems associated with monolithic ceramics. By incorporating high strength, relatively high modulus fibers into brittle ceramic matrices, combined high strength and high toughness composite materials can be obtained. Successfully manufactured ceramic matrix composites exhibit a high degree of non-linear stress-strain behavior with ultimate strengths, failure strains and fracture toughnesses that are substantially greater than that of the unreinforced matrix.

In order to exploit the benefits of fiber reinforcement in brittle ceramic matrices, it is well recognized that a relatively weak fiber/matrix interfacial bond strength is essential for preventing catastrophic failure from propagating matrix cracks. The interface must provide sufficient fiber/matrix bonding for effective load transfer, but must be weak enough to debond and slip in the wake of matrix cracking, leaving the fibers to bridge the cracks and support the far-field applied load. Fiber-reinforced ceramic matrix composites with very high fiber/matrix interfacial bond strengths (usually the result of chemical interaction during manufacture) exhibit brittle failure characteristics similar to that of unreinforced monolithic ceramics by allowing matrix cracks to freely propagate directly through the reinforcing fibers. Conversely, by reducing the interfacial bond strength, the fiber and matrix are able to debond and slip promoting the arrest and/or diversion of propagating matrix cracks at/or around the reinforcing fiber. Since crack inhibition/fracture toughness enhancement is the primary advantage of fiber-reinforced ceramic matrix composites, properly engineered fiber coating systems are thus key to the structural performance of these materials. Control of interfacial bonding characteristics between the fiber and matrix following manufacture and during service is typically provided by the use of applied fiber coatings.

Fiber-reinforced ceramic matrix composites produced by the chemical vapor infiltration (CVI) process are a particularly promising class of engineered high-temperature structural materials, which are now commercially available. The principal advantage of the CVI process approach for fabricating ceramic matrix composites as compared to other manufacturing methods (e.g., reaction bonding, hot-pressing, melt infiltration, or polymer impregnation/pyrolysis) is the ability to infiltrate and densify geometrically complex, multidirectional fibrous preforms to near-net-shape with a ceramic matrix of high purity and controllable stoichiometry without chemically, thermally or mechanically damaging the relatively fragile reinforcing fibers. In addition, because it is a relatively low temperature manufacturing process, high purity refractory matrix materials can be formed (deposited) at a small fraction of their melting temperature ( $\sim T_m/4$ ). Despite the many possible high-temperature ceramic matrix composite material systems, however, the number of practical systems is limited by the currently available reinforcing fibers. To date, the majority of high performance ceramic matrix composites produced have focused primarily on carbon and polymer-derived SiC (Nicalon and Hi-Nicalon) fiber reinforcement and CVI-derived SiC matrices.

Carbon fiber-reinforced silicon carbide (C/SiC) and silicon carbide fiber-reinforced silicon carbide (SiC/SiC) composites produced by CVI have been identified by the aerospace and propulsion communities (both in the United States and in Europe) as an important enabling materials technology for thermostructural applications demanding high strength and toughness at temperatures to 1650°C. The high purity CVI-SiC matrix is not readily attacked by either hydrogen-rich or oxidizing environments up to 1650°C, and resists oxidation by the formation of an adherent and protective oxide surface scale. Along with being chemically compatible with a variety of commercially available refractory fiber reinforcements, SiC possess excellent thermal

shock resistance due to its combination of very high thermal conductivity (50-100 W/m/K) and low thermal expansivity (4.5 ppm/ $^{\circ}$ C). These thermophysical attributes are especially attractive for advanced rocket propulsion thrust chamber and exhaust nozzle applications.

Uncooled, single-walled rocket thrust chambers and nozzle components have been  
5 produced from fiber-reinforced ceramic matrix composites by a number of manufacturers. Material systems for these applications have included carbon fiber-reinforced carbon, or carbon/carbon (C/C), C/SiC and SiC/SiC composites produced by various manufacturing techniques, including CVI. In all cases, these rocket propulsion devices were fabricated as a simple, single-wall shell construction and passively cooled by radiation.

Until now, actively-cooled reinforced ceramic matrix composite thrust chambers have neither been designed nor produced for various technical reasons, including the lack of conception of a practical design approach, the perceived high level of complexity for such a fiber-reinforced composite design, manufacturing difficulty, and high cost necessary to produce such a device.

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#### SUMMARY OF THE INVENTION

In the present invention, a process is described for the design and manufacture of actively-cooled fiber-reinforced ceramic matrix composite tubular shell structures for high-temperature applications with emphasis on converging-diverging thrust chambers for liquid rocket propulsion systems.

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Tubular shell structures are defined as any open-ended three-dimensional body with a central longitudinal axis wherein the body of the structure is enclosed by either curved surfaces, flat surfaces or combinations thereof.

Fiber-reinforcement is defined as any refractory fibers, either continuous or discontinuous, used for producing a fibrous preform texture, which are capable of withstanding a use temperature of at least 800°C in an atmosphere which is thermochemically compatible with that fiber without suffering fundamental chemical, physical or mechanical degradation.

- 5 Examples include carbon fibers, silicon carbide fibers, silicon nitride fibers, aluminum oxide fibers, etc.

A fiber preform is a fibrous texture defined as any assemblage of one or more reinforcing fiber types produced by weaving, braiding, filament winding, fiber placement, felting, needling, or other textile fabrication process.

Fiber preforming is a textile fabrication process by which the collimated multifilamentary fiber bundles (tows) are placed and maintained in a fixed position for purposes of controlling both their orientation and content within a given volumetric space. As such, the spatial arrangement of fibers is referred to as a preform architecture.

- The braided architecture is one of the simplest and lowest cost fiber preforms for 15 producing continuous fiber-reinforced axisymmetric, tubular structures. Although constructed of interlaced tows in a planar 2-dimensional arrangement similar to woven fabric, braiding offers the added flexibility of interlacing fiber in three (3) directions both axial and helical. The most common braid architecture consists of helical fibers interlaced at a prescribed bias angle and are termed biaxial braids. Fixed axial fibers can be inserted around the mandrel circumference in 20 nearly any desired fraction with respect to the helical “braiders” to produce an architecture reinforced in three (3) discrete directions and are termed triaxial braids. Triaxially braided preforms offer certain benefits over biaxial architectures, such as the ability to tailor material isotropy or increase axial properties. In most cases, increased axial properties are gained at the

cost of sacrificed circumferential “hoop” properties. However, the optimum braid architecture for a given application is usually designed and selected on the basis of the combined axial and hoop properties required. Triaxial preforms typically yield preforms with slightly lower fiber volume fractions than that of the biaxial braided architecture. This is due to the crimping and  
5 bunching of the fiber tows at the braid triple point, which results in increased braid layer thicknesses and larger internal voids.

Fiber coating is defined as any refractory composition of either carbon, metal carbide, metal nitride, metal boride, metal silicide, metal oxide, or combinations thereof which is (are) deposited (for example by chemical vapor infiltration) onto the refractory fibers either before or after fiber preforming for purposes of controlling the fiber/matrix interfacial bonding characteristics in the resultant composite. The resultant fiber coating thus encapsulates the reinforcing fibers. Examples include pyrolytic carbon, silicon carbide, silicon nitride, boron carbide, boron nitride, etc.; either as a single-layer phase, multilayered phase or as a phase of mixed composition.

Ceramic matrix is defined as any refractory composition of either carbon, metal carbide, metal nitride, metal boride, metal silicide, metal oxide, or combinations thereof which is subsequently deposited (for example by chemical vapor infiltration) onto the previously coated refractory fibers within the fibrous preform thereby encapsulating the fibers and consolidating the preform into the resultant densified composite. The reinforcing fibers of the fibrous preform  
20 thus become embedded within and supported by the surrounding matrix. Examples include pyrolytic carbon, silicon carbide, silicon nitride, boron carbide, boron silicide, etc., either as a single phase, multilayered phase or as a phase of mixed composition.

### BRIEF DESCRIPTION OF THE FIGURES

Figures 1 (A-E) show one embodiment of an actively-cooled fiber-reinforced ceramic matrix composite tubular structure comprising an axisymmetric rocket propulsion thrust chamber of converging-diverging geometry.

Figure 2 (A-B) show a two-piece inner tooling mandrel suitable for defining the inner wall geometry of a converging-diverging thrust chamber wherein the mandrel is split and joined at the minimum cross-section (e.g., throat) for easy removal and reusability.

Figure 3 shows one configuration of a single axial cooling channel tooling mandrel segment suitable for defining the internal cooling channel passage of an actively-cooled tubular structure of converging-diverging geometry.

Figure 4 (A-B) show a base cooling channel tooling mandrel of compound converging-diverging geometry prior to segmenting (Figure 4A) and following segmenting into a plurality of individual cooling channel tooling mandrel segments (Figure 4B).

Figure 5 (A-D) show the assembly configuration of the plurality of individual cooling channel tooling mandrel segments disposed about the inner tooling mandrel.

Figure 6A shows a fibrous preform overlaid onto an inner tooling mandrel of converging-diverging geometry.

Figure 6B shows a plurality of fibrous preforms overlaid onto the individual cooling channel tooling mandrel segments of converging-diverging geometry.

Figure 6C shows a fibrous preform sub-assembly of converging-diverging geometry comprising a plurality of preformed cooling channel tooling mandrel segments disposed about the preformed inner tooling mandrel.

Figure 7 shows a pair of fully completed fibrous preform assemblies constructed in accordance with the present invention shown with the inner tooling mandrel removed for illustration.

Figure 8 shows a pair of densified fiber-reinforced ceramic matrix composite rocket 5 propulsion thrust chamber structures of converging-diverging geometry shown prior to the removal of the plurality of embedded cooling channel tooling mandrel segments.

Figure 9 shows a pair of densified fiber-reinforced ceramic matrix composite rocket propulsion thrust chamber structures of converging-diverging geometry wherein the plurality of cooling channel tooling mandrel segments have been removed.

#### DETAILED DESCRIPTION OF THE INVENTION

Figure 1 shows one embodiment of an actively-cooled fiber-reinforced ceramic matrix composite tubular shell structure comprising an axisymmetric converging-diverging rocket propulsion thrust chamber **10**. It can be appreciated that other tubular shell geometries (e.g., cylindrical tubes, conical tubes, rectangular tubes, etc.) can be fabricated with internal cooling 15 channels without departing from the scope of the present invention. The basic structural arrangement of the actively-cooled thrust chamber **10** comprises three (3) fundamental sub-structural elements: an inner combustion chamber vessel wall **11**, an annular array of integral cooling channels **12** each separated by periodically spaced radial coupling webs **13**, and an outer primary pressure vessel wall **14**. The inner vessel wall **11** functions as a “hot” liner in contact 20 with and directing the flow of combustion gases produced during operation. The inner vessel wall **11** is preferably designed to be as thin as structurally possible and fabricated from a material possessing combined high thermal conductivity and high-temperature mechanical properties (e.g., strength, toughness and stiffness). A thin-wall inner shell **11** constructed of a high thermal

conductivity material is preferred in order to maximize the transfer of thermal energy from the hot inner wall **11** to the cooling fluid passed through the cooling channel passages **12** during operation. A material with high-temperature strength and toughness is preferred because of the extreme high-temperatures and pressures imposed on the inner liner **11** by the combustion products.

In order to provide for active cooling of the thrust chamber structure **10** shown in Figure 1, an annular array of embedded cooling channels **12** is provided. These channels **12** serve to contain and transport pressurized cooling fluid through the interior of the thrust chamber structure **10**. Because it is desirable for the thermally effective surfaces of the cooling channels **12** to be in intimate contact with the hot inner wall **11** for maximum cooling efficiency, a trapezoidal-shaped cooling channel passage **12** cross-sectional geometry as shown in Figure 1 is preferred in order to maximize the contact surface adjoining the inner wall **11** and the cooling fluid. It can be appreciated that other cooling channel geometries (e.g., circular) can be utilized for fabrication of the cooling channels without departing from the scope of the present invention.

However, the trapezoidal-shaped cooling channel geometry **12** is preferable for the present application over that of circular based on: (1) providing the maximum wetted surface area per unit channel packing, resulting in the most efficient convective heat transfer (e.g., cooling) characteristics; (2) the ability to continuously vary the cooling channel cross-sectional area so as to passively control local coolant flow rates for optimal thermal control; (3) providing the greatest structural integrity via direct, linear load paths from the inner vessel wall **11** to the outer vessel wall **14**; (4) minimizing channel-to-shell nesting voids **15**, thereby minimizing interlaminar stresses and diminished heat transfer efficiency; (5) the ability to easily integrate the inner shell wall **11**, outer shell wall **14** and cooling channel **12** components into a single

monocoque assembly **10**; and (6) the relative ease of manufacturing the geometrically-complex coolant channel tooling mandrels without the need for an elaborate and expensive multi-axis, numerically-controlled milling machine operation.

A plurality of discrete cooling channels **12** is preferred over an open annulus, dual-shell  
5 (e.g., cooling jacket) cooled structure design configuration. The array of individual cooling channel passages **12** aids to straighten the flow of the cooling fluid passed therethrough, thereby minimizing undesirable fluid mechanical effects (e.g., rotational flow), which can compromise the convective heat transfer efficiency of the heat exchanger.

The cooling channels **12** are separated from each other by periodically spaced radial web  
10 structures **13**. The plurality of radial webs **13** serve to structurally couple the inner **11** and outer  
14 vessel walls into a single integrated monocoque assembly **10**. The webs **13** provide a direct radial load path from inner wall **11** to outer wall **14**, which serves to increase the weight-specific structural efficiency of the overall thrust chamber vessel **10**. The webs **13** also provide discrete radial thermal conduction pathways (e.g., radial cooling fins), which serve to increase the effectiveness of the convective heat exchanger.  
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The actively-cooled structure **10** is fabricated from a fiber-reinforced ceramic matrix composite material specially designed for high-temperature structural applications, such as rocket propulsion thrust chambers, burner tubes, heat exchangers, and other severe environment thermostructural components. The ceramic matrix composite material comprises a fibrous preform composed of an assemblage of refractory fibers, a fiber coating material encapsulating the refractory fibers, and a ceramic matrix material that consolidates the coated fibrous preform into a densified composite.  
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A first step in producing a near net-shape actively-cooled fiber-reinforced ceramic matrix composite tubular shell structure **10** in accordance with the present invention is the fabrication of tooling mandrels. The tooling mandrels serve to provide structural support and geometric definition of the fibrous preform, which will ultimately define the geometry of the resultant composite structure following fiber coating, matrix densification and mandrel removal.

Suitable tooling mandrel materials should be selected on the basis of several considerations: (1) thermochemical stability in the fiber coating and matrix densification processes; (2) thermochemical and thermomechanical compatibility with the preform reinforcing fiber; (3) thermophysical stability for maintaining desired component geometry during and following materials processing; (4) adequate structural integrity for handling the fibrous preform textile fabrication processes and subsequent preform assembly; (5) ease of removal; and (6) cost. Examples of suitable materials include graphite, molybdenum and stainless steel.

As a generalized example, the inner mold line (e.g., inner surface) of an axisymmetric converging-diverging ceramic matrix composite thrust chamber **10** as shown in Figure 1 is produced and defined by an inner tooling mandrel. Because of its axisymmetry, this inner tooling mandrel can be fabricated using standard lathe turning techniques. Lathe machining of the converging-diverging chamber geometry from a suitable tooling material can be accomplished using a simple profile tracer template, numerical control, or other machining techniques well known in the art. Figure 2 shows an example of a converging-diverging split inner tooling mandrel **20** suitable for use in fabricating the inner shell wall **11** of an actively-cooled ceramic matrix composite rocket propulsion thrust chamber **10**.

For the example of a converging-diverging geometry, the inner tooling mandrel **20** can also be fabricated from two (2) segments, which can be split at the minimum cross-section **21**

(e.g., throat) and mechanically joined by an axial bolted fastener to facilitate easy removal and reusability of the inner tooling mandrel **20**. A simple actively-cooled cylindrical tube geometry on the other hand can be easily defined by a simple, single-piece cylindrical inner tooling mandrel of constant diameter along its length. Regardless of the final tubular geometry desired, 5 excess length at either end of the inner tooling mandrel **20** may be desirable to accommodate certain fiber preforming methods, such as braiding, and subsequent fibrous preform assembly as will be described. In this event, any irregular and/or excess material at the ends of the densified composite structure can be trimmed and discarded following completed fabrication.

Following construction of an inner tooling mandrel **20** for defining the inner wall **11** geometry of the ceramic matrix composite tubular shell structure **10** to be produced, a fibrous preform of predetermined architecture and thickness is constructed onto the inner tooling mandrel **20** by a suitable textile fabrication process, such as braiding. The inner fiber preform forms the inner shell wall **11** sub-structure of the actively-cooled composite structure **10**; thus, the preform architecture and preform thickness should be chosen with this design consideration 15 in mind.

For increased resistance to damage or distortion sustained by subsequent handling, the fiber preform can be restrained onto the inner mandrel by hoop wrapping the excess ends with a suitable reinforced tape or string, as these ends may eventually be trimmed away and discarded.

The inner mold line of the array of annularly arranged cooling channel passages **12** 20 within the composite structure **10** are produced and defined by individual tooling mandrel segments. The number of cooling channels passages **12**, their corresponding orientation (e.g., axial, helical or sinusoidal), and cross-sectional area properties are selected based on satisfying

the combined structural, thermal and fluid mechanical requirements of the actively-cooled structure as determined from design.

Once a suitable design configuration for the cooling channels has been selected, individual cooling channel mandrel segments are then fabricated in accordance to this selection.

5 Figure 3A shows an example of a single axial cooling channel mandrel segment **30** suitable for use in fabricating an actively-cooled fiber-reinforced ceramic matrix composite thrust chamber **10** of general converging-diverging geometry. Fabrication of a plurality of individual tooling mandrels **30** having the complex convoluted geometry shown in Figure 3 would normally require sophisticated (e.g., numerically controlled) multi-axis mill machining. Fabrication of helical or sinusoidally undulating channel tooling mandrels would be even more complex. The present invention, however, describes a greatly simplified manufacturing technique for fabricating the geometrically complex cooling channel mandrel segments **30** necessary for producing an actively-cooled ceramic matrix composite converging-diverging thrust chamber **10** or other enclosed tubular structure.

15 In the present invention, a “base” cooling channel tooling mandrel corresponding to the selected geometry of the cooling channel passages **12** to be produced within the structure is fabricated from a suitable tooling material using standard lathe turning and mill or saw cutting techniques. Selection of a suitable tooling material for the fabrication of the base cooling channel mandrel is important, as previously described, and must also take into consideration the process 20 to be used for subsequent removal of the individual tooling mandrel segments, which may become embedded or intimately attached following the matrix densification of the actively-cooled ceramic matrix composite structure. As discussed below, graphite is a suitable choice for fabrication of the base cooling channel mandrel as it may be subsequently removed using

conventional techniques (e.g., bead blasting) without damaging the surrounding ceramic matrix composite structure. Other tooling materials may also be appropriate and are discussed below.

The base cooling channel tooling mandrel for an axisymmetric converging-diverging cylindrical geometry (e.g., actively-cooled thrust chamber) is fabricated by machining the outer diameter of the selected tooling material with a desired profile, which does not necessarily have to correspond to the profile of the inner mandrel **20** previously described. Following machining of the outer profile, an inner profile is then produced onto the mandrel which directly corresponds to the geometric profile of the inner mandrel **20**. The difference between the inner and outer diameters of the base cooling channel mandrel ultimately defines the cooling channel passage cross-sectional area as will be described below. Figure 4A shows a suitable base cooling channel mandrel **40** for producing the individual cooling channel mandrels **30** of a converging-diverging thrust chamber structure **10** as described above. With reference to Figure 4A, the resultant base cooling channel mandrel **40** for a converging-diverging thrust chamber **10** comprises a hollow tubular shell structure having compound inner and outer converging-diverging profiles. As previously described, the inner and outer profiles of the base cooling channel mandrel **40** can be independent of one another and will thus form the desired cross-sectional geometry of the internal cooling passages **12**. The inner profile of the base mandrel **40** is designed to geometrically conform to the profile of the inner mandrel **20** with prescribed radial accommodation (e.g., relief or gap) for the thickness of fiber preforms, which are ultimately produced over the respective inner mandrel **20** and individual cooling channel mandrels **30** following their fabrication. Thus, the radial dimensions of the inner mandrel **20** and the internal contour of the base mandrel **40** differ only by the fiber preform thickness desired along the length of the respective tools, and ultimately in the completed composite structure.

Following fabrication of the inner and outer profiles, the compound converging-diverging base mandrel **40** as shown in Figure 4A is then cut into the desired axial, helical or sinusoidal segments using conventional cutting saw or vertical milling machining techniques. The resultant cut segments thus produce the individual cooling channel mandrels **30** which are used to form  
5 the internal cooling channel passages **12** of the actively-cooled ceramic matrix composite structure **10**. Figure 4B shows a converging-diverging base cooling channel mandrel **40** that has been cut axially into individual cooling channel tooling mandrel segments **30**.

The cutting process for fabricating the individual cooling channel mandrel segments **30** from the base mandrel **40** is preferably performed by using a prescribed kerf, or cut width in order to control the desired thickness of the fibrous preform that, as described below, will eventually be produced onto and formed by the individual cooling channel tooling mandrel segments **30**. The appropriate kerf width for cutting the cooling channel mandrel segments **30** from the base mandrel **40** should be approximately twice the desired fibrous preform thickness to be produced on each channel mandrel. This is to accommodate the cumulative preform thickness build-up from adjoining cooling channel mandrel segments upon their assembly and compaction.  
15 The cumulative preform thickness between each adjoining cooling channel fiber preform following their annular assembly forms the array of radial coupling webs **13** of the actively-cooled ceramic matrix composite structure **10**.

For axially oriented channels **12**, the individual cooling channel mandrel segments **30** are  
20 produced simply by a systematic process of longitudinal cutting followed by rotational indexing of the base mandrel **40** at discrete angular intervals. The angular indexing interval ( $360^\circ/n$ ) is selected based on the number of like cooling channels ( $n$ ) desired. This systematic process of cutting and indexing is repeated until the entire base mandrel shell **40** is cut into the

desired number of equal segments **30**. Figure 4B shows an example of a plurality of axially-oriented cooling channel mandrel segments **30** produced from a single compound converging-diverging base cooling channel mandrel **40** as is shown in Figure 4A.

For helically oriented channels, the individual cooling channel mandrel segments may be produced by simultaneously rotating the base cooling channel mandrel **40** about its longitudinal axis while cutting. The rate of angular rotation combined with the rate of longitudinal cutting is synchronized to produce the desired helical cut path. Following each helical cut, the base cooling channel mandrel **40** is rotationally indexed as previously described and the cutting process is repeated in manner similar to that of axially aligned cooling channel mandrel segments **30**.

For sinusoidally undulating channels, the individual cooling channel mandrel segments are produced by simultaneously undulating the base cooling channel mandrel **40** about its longitudinal axis in a cyclic rotational or back-and-forth motion while cutting. The rate of the rotational reversal while cutting ultimately defines the amplitude of the undulating individual cooling channel mandrel segments produced. The rate of longitudinal cutting is thus synchronized to produce the desired sinusoidal period, or wavelength. Following each sinusoidal cut, the base cooling channel mandrel **40** is rotationally indexed and the cutting process is repeated in a manner similar to that of axially aligned cooling channel mandrel segments **30**.

To aid in ease of fabricating the cooling channel tooling mandrel segments, it is best not to fully cut the entire length of the base mandrel **40** during segmenting of the individual cooling channel mandrels **30** as the remaining uncut base mandrel **40** becomes flexible and awkward to handle and accurately index during machining. By retaining short uncut lengths at both ends of the base mandrel **40** during cutting, the base mandrel **40** remains circumferentially rigid and adequately stabilized by the remaining uncut portion of the attached individual cooling channel

mandrel segments **30**. After completion of cutting all cooling channel mandrel segments **30**, the uncut ends of the base cooling channel mandrel **40** are then cut perpendicular to the longitudinal axis of the base mandrel **40** and discarded, thereby freeing all the individual cooling channel mandrel segments **30**. The final length of the individual cooling channel mandrel segments **30** is therefore shortened by the length of the discarded trim material. Thus, the base mandrel **40** can be made longer to accommodate this subsequent end trimming method. As-machined sharp edges on the individual cooling channel mandrel segments **30** can be removed by chamfering if necessary. Chamfering aids in minimizing potential fiber damage from abrasion during fiber preform fabrication. Although beneficial for fiber preforming, excessive chamfering can cause excessive preform nesting assembly voids **15** in the resultant composite structure **10**.

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Figure 5 shows the assembly form of individual cooling channel mandrel segments **30** disposed around an inner tooling mandrel **20** for producing an actively-cooled ceramic matrix composite thrust chamber **10** of converging-diverging geometry in accordance with the present invention (axial cooling channel design shown without fibrous preform for illustration). As shown in Figure 5, a predetermined machining kerf has been chosen to allow for circumferential relief (e.g., gaps) between each adjacent cooling channel mandrel segment **30** in order to form the radial coupling webs **13**. Radial relief between the inner surface of the individual cooling channel mandrel segments **30** and the adjacent inner tooling mandrel **20** is also provided in order to form the inner vessel wall **11**. It is important that these relief features be designed in accordance with the selected thickness of the fibrous preforms subsequently produced onto the individual cooling channel mandrel segments **30** and the inner tooling mandrel **20** for proper assembly. For example, a kerf of greater than twice the preform thickness on the individual cooling channel mandrel segments **30** will result in producing a composite with excessive

nesting voids **15**, whereas a kerf of less than twice the preform thickness on the individual cooling channel mandrel segments **30** will result in undesirable assembly interference. Significant assembly interference can result in producing a composite with either insufficient inner wall fiber volume fraction or delamination between the inner wall **11** and adjoining cooling channels **12**, which can significantly diminish both the structural and thermal performance of the resultant actively-cooled ceramic matrix composite structure **10**.

Following cooling channel mandrel fabrication, a fibrous preform of predetermined architecture and thickness is then produced onto each of the individual mandrel segments **30** by a suitable preform fabrication process, such as braiding.

Having an overlaid fiber preform, the individual cooling channel tooling mandrel segments **30** are then assembled around the perimeter of the inner tool mandrel **20** in an annular array. As described previously, the inner tooling mandrel **20** has already been overlaid with its own inner fibrous preform. The individual cooling channel mandrel segments **30** are preferably held into position and compacted against the inner fibrous preform radially and against themselves circumferentially to obtain the desired preform thickness in the inner wall **11** and radial webs **13**, respectively. Control of these thicknesses via tooling design (e.g., preform relief), fiber preforming and preform assembly ultimately controls the fiber volume and nesting void fraction within the resultant composite structure. Restraint of the compacted channel fiber preform segments against the inner fiber preform may be accomplished by tightly hoop wrapping the ends of the channel mandrel segments with a suitable reinforced tape or string. It is preferable to restrain the tooling/preform sub-assembly in the end region of excess length as these ends can be trimmed away and discarded following preform consolidation and matrix densification processing.

Figure 6A shows the inner tooling mandrel **20** having an overlaid fibrous preform **60**, which forms the reinforcement of the inner wall **11** of the ceramic matrix composite structure **10**. Figure 6B shows a number of cooling channel mandrel segments **30** having overlaid fibrous preforms **61** which form the reinforcement of the individual cooling channels **12** of the ceramic matrix composite structure **10**. Figure 6C shows the assembly of individual cooling channel mandrel segments **30** having overlaid fibrous preforms **61** disposed around the inner tooling mandrel **20** and inner fibrous preform **60**. Compacting and securing the individual cooling channel fibrous preform segments **61** against the inner fibrous preform **60** as a fibrous preform sub-assembly **62** is provided by hoop-wrapping **63** the excess ends of the individual cooling channel mandrel segments **30**.

Once the fiber preform sub-assembly **62** has been fabricated, it is then overlaid with an outer fibrous preform by a suitable textile fabrication process, such as braiding. The outer fibrous preform will thus conform to the geometry of the underlying tooling/preform sub-assembly **62**. Figure 7 shows a completed converging-diverging thrust chamber tooling/fibrous preform assembly **70** (inner tooling mandrel **20** removed for illustration) ready for subsequent fiber coating and matrix densification. For ease of handling in preparation for subsequent fiber coating and/or matrix consolidation processing, the entire fibrous preform assembly **70** can also be restrained by hoop wrapping the excess ends with a suitable reinforced tape or string **72**, again, as these ends can eventually be trimmed away and discarded. The outer fibrous preform **71** forms the outer wall **14** of the actively-cooled ceramic matrix composite structure **10** produced. Thus, the fabrication of the fibrous preform **70** corresponding to the desired actively-cooled tubular structure **10** to be produced is completed following the deposit of the outer fiber preform **71** onto the underlying fibrous preform sub-assembly **62**.

Following fabrication of the fibrous preform assemblage **70**, a fiber coating or fiber coating system is deposited (for example by chemical vapor infiltration) onto the entire tooling/preform assembly **70**. If the reinforcing fibers have been coated prior to preforming, this processing step may not be necessary. As previously described, the fiber coating serves to control the desired fiber/matrix interfacial compliance and bonding characteristics in the resultant ceramic matrix composite structure produced. Examples of suitable fiber-coating materials include pyrolytic carbon, silicon carbide, silicon nitride, boron carbide, boron nitride, etc., either as a single layer phase, multilayered phase or as a phase of mixed composition.

Following the application of the fiber coating or fiber coating system, a ceramic matrix is deposited (for example by chemical vapor infiltration) onto the entire tooling/preform assembly **70**. The process of matrix deposition using chemical vapor infiltration is well known in the art and is also described in U.S. Patent No. 5,455,106 and Applicant's co-pending patent application having Attorney Docket Number 260/175, the disclosures of which are incorporated herein by reference in their entirety. It can be appreciated that other matrix processing techniques (e.g., polymer impregnation/pyrolysis, melt infiltration, etc.) can be utilized for densification of the fibrous preform assembly **70** without departing from the scope of the present invention.

Depending on the method of matrix deposition processing employed, consolidation of the fibrous preform assembly **70** can be performed in two sequential steps. The first processing step involves the application of a small volume fraction of matrix to stiffen or rigidize the fibrous preform assembly **70**. A properly rigidized fiber preform assembly **70** lacks flexibility and is sufficiently stiff enough to be handled and further processed without the aid of external fixturing and/or tooling for stabilizing and maintaining the desired component geometry. The amount of deposited matrix necessary to adequately rigidize the preform assembly **70** is dependent on the

matrix composition and processing method employed. Following initial rigidization, the inner tooling mandrel **20** may then be removed to enable consolidation to commence on the freestanding fibrous preform assembly **70** as shown in Figure 7. Early removal of the inner tooling mandrel **20** following initial rigidization facilitates easy mandrel extraction in addition to 5 mandrel reusability and enhanced matrix densification processing economics. However, for complex geometries (e.g., converging-diverging) and/or helical or sinusoidal cooling channel designs, the individual cooling channel mandrel segments **30** must remain captive within the fibrous preform assembly **70** due to their convoluted geometry. Should straight axial cooling channel mandrel segments be chosen, such as that suitable for actively-cooled right cylindrical or 10 tapered cylindrical tubes, these mandrels may be effectively removed at this step, as they may not be geometrically captured within the preform. Thus, removal of the individual cooling channel mandrel segments is dependent on the geometry of the cooling channel mandrels chosen which, is dependant on the application for which the actively-cooled ceramic matrix composite structure is intended.

15 The second processing step following the removal of the inner mandrel **20** is that of preform consolidation. Consolidation of the rigid, yet still highly porous preform assembly **70** with a ceramic matrix is thus performed until a zero or near-zero residual open porosity level is attained in the resultant composite. The reinforcing fibers of the inner fibrous preform **60**, outer fibrous preform **71**, and cooling channel fibrous preforms **61** thus become embedded within, 20 supported by and fused to the surrounding matrix. Examples of suitable matrix materials include pyrolytic carbon, silicon carbide, silicon nitride, boron carbide, boron nitride, etc., either as a single-layer phase, multilayered phase or as a phase of mixed composition.

Following matrix densification processing, the densified actively-cooled ceramic matrix composite structure is prepared for either rough or final detailed machining and cooling channel tooling mandrel removal. Rough machining involves removing the excess material length used initially for assembling and restraining the fibrous preform from both ends of the actively-cooled 5 ceramic matrix composite tubular structure. Rough machining/trimming can be performed using standard diamond cutting saw machining techniques. The trim end remnants are thus removed from the densified ceramic matrix composite structure and discarded. Figure 8 shows a near-densified ceramic matrix composite thrust chamber structure of converging-diverging geometry which has undergone rough machining. Cutting off the ends of the actively-cooled ceramic matrix composite structure, as shown in Figure 8, exposes the ends of the embedded cooling channel mandrel segments **30**, which is necessary for their removal in the case of cooling channels having convoluted geometry (e.g., converging-diverging).

The method of removing the embedded cooling channel mandrels segments **30** is dependent on the tooling material employed for fabrication of the base cooling channel mandrel 10 **40**. Graphite cooling channel mandrels **30** can be removed by either oxidation or grit blasting. For composite materials not composed of carbon or other low oxidation threshold constituents (e.g., reinforcing fiber, fiber coating or matrix) oxidative combustion of graphite mandrels is performed by thermally conditioning the composite structure in a furnace heated to at least 15 425°C in an ambient (air) environment. The rate of mandrel removal can be accelerated with increased conditioning temperatures and/or increased oxygen partial pressures. Conditioning temperatures and oxidizing atmospheres should be maintained well under that necessary to cause 20 thermochemical (e.g., oxidation) or thermomechanical degradation of the ceramic matrix composite material.

For ceramic matrix composite materials of sufficient erosion resistance (e.g., high hardness and strength), grit blasting is a viable alternative to oxidative combustion for rapid removal of graphite cooling channel mandrels **30**. Because of its combination of low hardness and strength, bulk graphite exhibits very low resistance to hard particle erosion. Blast media and delivery pressures are selected based on providing the most aggressive erosive removal of the mandrels without imparting erosive damage or compositional contamination to the adjacent composite material. Small diameter orifice nozzles combined with nozzle extensions can be used to enable access down the otherwise long, slender and convoluted channel passages **12**.

Metallic cooling channel mandrels **30** can be effectively removed by acid digestion. For ceramic matrix composite materials composed of constituents which are chemically compatible with suitable etching acids (e.g., nitric acid), dissolution of metallic cooling channel mandrel segments **30** is performed by submerging the composite structure into an acid bath. The rate of mandrel removal can be accelerated with increased acid bath temperatures and/or increased acid concentrations or aggressive acid mixtures. For very long and slender channel passages **12**, cyclic pressure or vacuum pulsing of a digestive bath system aids in the accelerated removal of high aspect ratio cooling channel mandrel segments **30**.

Following removal of the embedded cooling channel mandrels **30**, final detailed machining is performed on the densified ceramic matrix composite structure to satisfy specific geometric and dimensional tolerance requirements (e.g., parallelism and flatness of the free ends, chamfering or beveling of sharp edges, and/or lathe turning of the inner and/or outer diameter(s) to incorporate provisional details for coolant manifold integration, etc.). Detailed machining is typically performed using standard diamond grinding techniques.

Following removal of the individual cooling channel mandrel segments **30** and final machining, the actively-cooled ceramic matrix composite structure **10** is preferably processed with a final matrix seal coating. Seal-coat processing as a final step in the fabrication of the actively-cooled ceramic matrix composite structure serves to over-coat the freshly exposed interior walls of the cooling channel passages **12** as well as providing an environmentally protective coating on free-edge surfaces freshly exposed from cutting and final machining.

Figure 9 shows a completed actively-cooled fiber-reinforced ceramic matrix composite thrust chamber structure **10** of converging-diverging geometry.

#### EXAMPLE

The present invention will now be described with reference to a specific example comprising the fabrication of an actively-cooled fiber-reinforced ceramic matrix composite thrust chamber of converging-diverging geometry using the methods previously described. For this example, a simple axially oriented cooling channel design is selected for purposes of demonstrating the manufacturing methods of the current invention. The example thrust chamber is produced with a 50.8 mm diameter cylindrical combustion chamber, a 25.4 mm throat, a 101.6 mm diameter conical exhaust (expansion) nozzle, and is 203.2 mm in overall length. The convergent and divergent sections are of simple conical geometry, both with a half angle of 20-degrees with respect to the longitudinal axis. The ceramic matrix composite structure is designed to safely sustain combustion chamber pressures and coolant delivery pressures of up to 7.5 Mpa and 11.2 Mpa, respectively. The axial heat exchanger circuit is designed to reduce the combustion chamber inner wall temperature from over 3,600°C to less than 1,650°C using

cryogenic liquid hydrogen coolant delivered at a temperature of -220°C and flow rate of 0.45 kg/sec.

The material system selected for demonstrating the actively-cooled thrust chamber design and manufacturing methodology is a braided Hi-Nicalon silicon carbide (SiC) fiber-reinforced

- 5 CVI-SiC ceramic matrix composite (SiC/SiC). The SiC/SiC composite material system is selected for its high thru-thickness thermal conductivity, negligible thru-the-thickness permeability, and inherent oxidation resistance. Continuous Hi-Nicalon SiC fiber is selected as the structural reinforcement for its excellent thermal expansion compatibility with the CVI-derived SiC matrix and adequate high-temperature mechanical properties. A braided preform architecture is selected because of its amenability for producing high-performance axisymmetric tubular structures and tailorable in-plane (e.g., axial and circumferential) mechanical properties.

Tooling mandrels (e.g., inner mandrel and cooling channel mandrel segments) for the example thrust chamber are fabricated from fine-grain, high-density bulk graphite. Bulk graphite is selected as the tooling material for its outstanding compatibility with the CVI-based PyC and SiC processes and low cost. The example thrust chamber design requires eighteen (18) individual channel mandrel segments, each sectioned at 20-degree intervals. All tooling mandrels are fabricated with adequate additional trim length for preform fabrication, assembly, compaction, and restraint for reasons previously described.

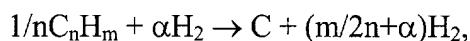
- For this example, a biaxially braided Hi-Nicalon SiC fiber preform is selected for its  
20 combination of high volumetric loading of fiber reinforcement, excellent hoop properties and overall design simplicity. The fibrous preform is produced by braiding four (4) fabric-like layers in succession onto the inner split mandrel and four (4) fabric-like layers onto each of the eighteen (18) cooling channel mandrel segments. The selected braid bias (helix) angle of the fiber

reinforcement in each preform layer is approximately  $\pm 60$ -degree with respect to the thrust chamber longitudinal axis. The  $\pm 60$ -degree fiber architecture is selected for its near-optimum balance of axial and hoop mechanical properties desired for axisymmetric composite pressure vessel structures.

5 Following the fiber preforming process, the eighteen (18) channel segments are disposed around and assembled onto the inner mandrel in an annular array. The assemblage is radially compacted and secured using a tight wrap of heavy-duty carbon yarn. Figure 6 shows a photograph of the braided inner mandrel, nine (9) of the eighteen (18) required cooling channel mandrel segments and the resultant sub-assembly of the respective fibrous preforms. The entire tooling/preform sub-assemblage is then overlaid with six (6) additional  $\pm 60$ -degree braid layers, thereby completing the actively-cooled thrust chamber fibrous preform assembly.

10 The initial CVI processing step is the application of a suitable fiber coating onto the fibrous preform assembly. As previously discussed, a fiber coating is required to impart the necessary fiber/matrix interfacial mechanical characteristics (e.g., low interfacial shear strength) 15 to promote high strength and toughness in the resulting composite. The selected fiber coating for this example is a two-layer, duplex coating system comprised of a first  $\sim 0.4 \mu\text{m}$  debond layer of pyrolytic carbon (PyC) followed by a second  $\sim 0.6 \mu\text{m}$  oxidation-barrier layer of boron carbide (B<sub>4</sub>C) produced by CVI methods.

15 The CVI-derived PyC fiber coating is produced in a high-temperature, low-pressure 20 chemical vapor infiltration (CVI) reactor by the thermal decomposition of a hydrocarbon-containing gas in the presence of hydrogen according to the following chemical reaction:



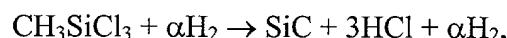
where  $C_nH_m$  is the gaseous hydrocarbon reactant (e.g., methane, propane, propylene etc.) and  $\alpha$  is defined as the molar ratio of  $H_2$  to  $C_nH_m$ . At deposition temperatures between 1000-1400°C, the deposit is typically smooth laminar PyC, which has a hexagonal structure and, depending on the deposition temperature, has a density ranging between 1.8-2.0 g/cm<sup>3</sup>.

5 The next processing step is the application of the outer  $B_4C$  oxidation-barrier coating produced by the CVI co-deposition of boron (B) from boron trichloride ( $BCl_3$ ) and carbon (C) from a suitable hydrocarbon gas using hydrogen as a carrier according to the following chemical reaction:



10 where  $4n$  is the molar ratio of  $BCl_3$  to  $C_nH_m$ , which is controlled to produce a near-stoichiometric  $B_4C$  coating composition at a given processing condition. At deposition temperatures between 1000-1300°C, the deposit is typically crystalline  $B_4C$ , which has a rhombohedrel structure and a theoretical density of 2.52 g/cm<sup>3</sup>.

15 The next CVI processing step is the rigidization of the fibrous preform with a ceramic matrix. The selected matrix constituent for this example is SiC for reasons previously discussed. The CVI SiC matrix is produced by the thermal decomposition of vaporized methyltrichlorosilane (MTS) using hydrogen as a carrier gas at elevated temperature and reduced pressure according to the following chemical reaction:



20 where  $\alpha$  is defined as the molar ratio of  $H_2$  to  $CH_3SiCl_3$ . At deposition temperatures between 900-1300°C, the deposit is typically crystalline beta-SiC, which has a cubic structure and a theoretical density of 3.21 g/cm<sup>3</sup>.

Following the initial deposition of the SiC matrix, the rigidized fiber preform is prepared for removal of the inner split mandrel prior to complete matrix densification processing. The CVI-derived SiC matrix typically bridges between the fibrous preform in physical contact with the adjacent inner tooling mandrel. This results in a slight, but easily frangible bonding of the  
5 fibrous preform to the adjacent mandrel. Removal of the split mandrel is performed by first removing the threaded axial tie bolt from the inner mandrel assembly. The SiC coating bond is then breached from the surrounding preform and inner mandrel and the opposing tools are then extracted from their respective ends of the preform assembly. Figure 7 shows a photograph of the rigidized thrust chamber preform with the inner split mandrel removed.

Consolidation processing of the free-standing fibrous preform with the SiC matrix is then performed until a near-zero residual open porosity level is attained in the composite.

After achieving near-final densification, rough machining is performed by trimming the excess material length from both ends of the ceramic matrix composite thrust chamber component using a diamond-bladed cutting saw. Removal of the excess material exposes the  
15 ends of the individual graphite cooling channel mandrel segments, which remain captive within the dense composite structure. Figure 8 shows a photograph of the densified ceramic matrix composite thrust chamber following removal of the trim ends.

Since the selected SiC/SiC composite is much harder and far more erosion-resistant than the selected graphite tooling material, the embedded cooling channel mandrel segments are  
20 easily removed by grit blasting methods. Commercially available borosilicate glass bead media provides adequate erosive qualities for removing the embedded graphite mandrels without inducing damage or contamination to the surrounding ceramic matrix composite material. Media delivery pressures of up to 0.7 MPa are satisfactory for the rapid removal of the graphite

mandrels without damaging the underlying ceramic matrix composite material. Grit blasting is best performed using a long, slender tubular nozzle extension to enable unobstructed access through the narrow convoluted cooling channel passages of the structure.

After complete removal of the graphite channel tooling mandrels, the ceramic matrix

5 composite thrust chamber is further processed with an additional SiC matrix infiltration/densification step. This processing step serves, in part, to densify and coat the freshly exposed interior walls of the cooling channel passages. Matrix processing is thus performed until a zero residual open porosity level is attained in the resultant composite. A zero open porosity level in the composite represents the maximum degree of densification achievable by CVI processing methods, as the chemical reactants no longer have diffusional access to the interior of the material via open porosity.

Detailed machining is then performed on the fully densified composite thrust chamber as necessary to satisfy specific geometric and dimensional tolerance requirements. Such requirements may include parallelism and flatness of the free ends, chamfering or beveling of sharp edges, and/or turning of the inner and/or outer diameter(s) to incorporate provisional details for external coolant manifold integration.

The final processing step in the manufacture of the actively-cooled ceramic matrix composite thrust chamber is that of CVI SiC “seal coating”. Seal coat processing as a final step in the fabrication of the composite thrust chamber serves to provide an environmentally-  
20 protective SiC over-coat to all surfaces exposed from detailed machining, as previously described. Figure 9. shows a photograph of a fully completed actively-cooled SiC/SiC composite thrust chamber produced by the manufacturing methods described herein.